Original Article

Evaluation of plasma nitriding process in the wear resistance of 7075 aluminum alloy

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Abstract

AlZnMgCu alloys are widely used in aerospace industry owing to their high mechanical strength, low density and good formability. However, these alloys have low hardness surface and chemical stability. The plasma nitriding is an alternative process of raising the tribological behavior of the materials. Thus, the main purpose of this paper is to investigate its effect in wear resistance of 7075 aluminum alloy. The 7075 – T6 aluminum alloy were nitrided via direct current plasma at temperatures ranging from 300 to 450 °C and pressure ranging from 0.6 to 0.9 Torr. The influence of nitriding process parameters such as gas mixture, pressure, temperature, and treatment time on the characteristic of nitrided layer were analyzed using X-ray diffraction (XDR), mechanical profilometry and scratch test. The results showed that aluminium nitride (AlN) layers covered totally the substrates, and suggest that, among all treatment conditions explored in this work, the nitriding condition at 450 °C, 0.9 Torr, for 180 minutes was the most satisfactory, because the layer produced is less roughness and presents lower friction coefficient and higher critical load (LC).

Keywords: Plasma nitriding; Aluminum alloy; AlN; AA7075; Scratch test.

1 Introduction

The 7075 aluminum are widely used in the parts manufacture and components for airplan mainly due to their high levels of mechanical resistance compared to the other aluminum alloys. The high mechanical resistance is achieved throught the artificial aging process, called T6 treatment, and effectively depends on the type, morphology, volume fraction and size of the strengthening precipitates [1-3]. However, the 7XXX series alloys has low surface hardness, wear resistance and thermal and chemical stability, factors very relevant in fatigue life. An alternative to minimize such problems is nitriding process, since it is widely known that this process lead to an improvement in tribological properties of many metallic alloys [2]. Interesting properties of aluminum nitride (AlN), such as high hardness (HV 1400), and wear resistance, as well as better resistance to chemical corrosion, high thermal conductivity (comparable to Al) and electrical resistivity are very attractive for advanced technological applications [4-7].

Plasma nitriding is successfully used for hardening of the steel surface [8,9]. This occurs through luminescent discharge which is responsible for the ionization of the nitrogen gas and the acceleration of this ions against the surface of the material. The incorporation of N species into the metallic alloy is then driven by thermal diffusion. This process can produce nitrides of high hardness in combination with the elements present in the material. In the case of Al alloys, the nitriding processes has an additional challenge represent by the presence of a native thin layer of aluminum oxide (Al_2O_3) on the surface of Al, which prevents the diffusion of nitrogen in the material [8].Therefore, it is essential to remove the oxide layer by a sputtering pretreatment, as shown by Arai et al [10]. After cleaning, it is necessary that the oxygen (O_2) concentrations be maintained at very low levels to minimize Al_2O_3 reformation before the nitriding process.

Some authors have reported successfully nitride aluminum alloys but all with different parameters and different results.

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For instance, Moradshahi et al [6] studied the DC plasma nitriding process of pure Al (1100) and 2025 aluminum alloy, and had reported the formation of a dense and continuous AlN layer with thickness about 2.2 µm and nitrogen content about 35-40 at.%. Visuttipitukul et al. [11] had reported a 10 µm thickness layer on an aluminum-titanium alloy produced after 20h at 550 °C by DC plasma. Renevier et al. [7] showed that an aluminium nitride layer approximately 7 mm thick can be realized after for 15 h at 450 °C using the arc-assisted nitriding process, that is a configuration of plasma nitriding, and reached the conclusion that wear properties of aluminium are improved by such a plasma-assisted nitriding treatment. However, there are few papers published which describe the formation of AlN layers on aluminum and aluminum alloy substrate, which makes relevant the study of the plasma nitriding process of aluminum and aluminum alloys.

The objective of this study is to evaluate the influence of nitriding parameters including gas mixture, gas pressure, temperature and treatment time on AlN layer properties, such as strength, wear resistance, uniformity and homogeneity.

2 Materials and methods

The 7075-T6 aluminum alloy was supplied in the shape of cylindrical bars with a diameter of 15.88 mm. The chemical composition of the AA7075-T6 alloy obtained via optical emission analysis was 89.99 wt% Al, 5.4 wt% Zn, 2.4 wt% Mg, 1.7 wt% Cu, 0.19 wt% Cr, 0.16 wt% Fe and 0.16 wt% Mn.

The plasma nitriding process was performed in a 20-litres cylindrical stainless steel vacuum chamber. Figure 1 shows the main details of the system. A thermocouple attached to the cathode was used to control nitriding temperature.

A mechanical pump is coupled to the system to ensure control of the pressure during the nitriding process, and electronic mass flow controllers were used to regulate and measure the gas flow rate.

The surface of the specimen was manually polished and then ultrasonically cleaned with acetone for 20 minutes prior to the nitriding process. Then, they were introduced into the reactor and vacuum was performed for 30 minutes, until the residual pressure achieves 1.10^{-5} Torr. Then a mixture of argon (30 sccm) and hydrogen (30 sccm) gases are admitted into the chamber and a pre-heating process is done by turning on plasma for approximately 2 hours depending on the desired temperature for nitriding. This process is also important in order to perform the sputtering of the oxide layer.

Upon reaching the nitriding temperature, the nitrogen is admitted into the chamber (with plasma on) thus initiating the nitriding process. Nitriding process were carried out with a temperature and pressure varying between 300-450 °C and 0.6 - 4 Torr, respectively. Table 1 shows the main process parameters used in our nitriding process. After the nitriding process the samples were vacuum cooled in nitrogen gas flow.

X-ray diffraction (XRD) analysis was used to evaluate the nitride layer structure and the possible formation of AlN. This analysis was performed by a diffractometer Rigaku — Ultima IV, by using a CuK-α radiation produced under 40 kV and 30 mA. Diffraction spectra were collected by scanning 2θ from 30° to 85°, with a 1° fixed grazing angle.

Profilometry analysis were performed using a mechanical profile KLA Tencor P7. The tests were conducted in triplicate.

The nitrided layer resistance was analysed using a nano tribometer on the scratch test mode in a Anto Paar equipament by using a diamond tip, according to ASTM C1624. A progressive normal load was applied from 30 mN to 10.000 mN for 1- 1.5mm.

3 Results and discussion

3.1 Nitriding condition

Due to the scarcity of nitriding studies in 7075 aluminum alloy, the determination of the experimental parameters

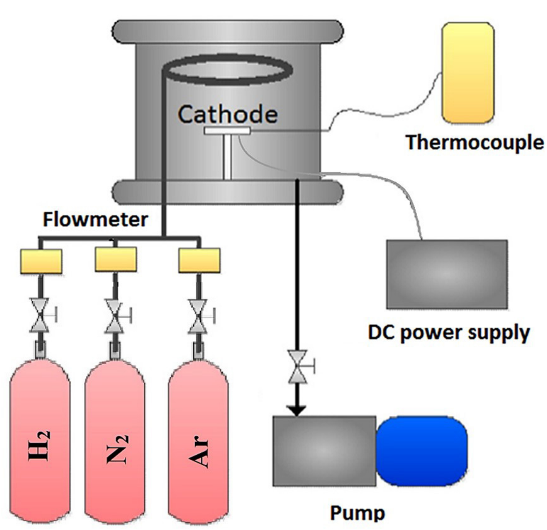

Figure 1. Schematic drawing of nitriding system – adapted [12].

(time, temperature, pressure and gas flow) for the initial nitriding tests was based on nitriding study in other aluminum alloys [6-8,13-15]. Therefore, some conditions of nitriding process were not successful. Samples 1, 2 and 4 (see Table 1); melted during nitriding process, indicating that pressure of nitriding chamber can not exceed 1 Torr and the increase of the power, which causes in the increase of temperature, must be slow in order to avoid formation of electric arcs on the sample surface, which increase the surface temperature and can cause the fusion of this region. Samples 3, 5, 6, 7, 8 and 9 presents a dark coloring characteristic of substrates successfully nitrided, as already observed by others researchers [15]. The color of the nitrided samples was darker for higher temperature and longer treatments, as a result of the increase in occurrence of reactions between aluminum and nitrogen governed by diffusion processes, leading to increased aluminum nitride formation, as already verified by some researchers [16,17]. The condition 3, which had the lowest temperature and pressure, had the lighter coloration, and condition 9, that has the highest temperature and one of the highest pressures, presented the darker coloration, which suggests a higher formation of aluminum nitride due to the greater diffusion process.

3.2 X ray diffraction

Several experimental studies report the existence of two different structures of AlN layers: hexagonal compact (HC) and cubic face centered (FCC) [6,12,15-18]. Diffraction peaks correspond to AlN with FCC structure were detected, as can be observed in the X-ray diffraction (XRD) peaks shown in Figure 2.

3.3 Roughness

The results of the analysis by mechanical profilometry of the non-nitrided substrate and the nitrided samples are presented in Table 2, together with the respective average roughness.

Due to the nitriding process being a diffusional process, where the formation of nitride layer and the maintenance of nitriding temperature occur by the ions collision on the substrate surface, there was a significant increase in the roughness in all nitriding conditions with respect to the non-nitrided sample as expected. It was observed that nitriding treatment conditions 3, 6 and 9 were the most satisfactory for obtaining AlN layers with less roughness. It is worth mentioning that the surface finish becomes an important factor in the life of a material subjected to fatigue, due to the possible presence of tension concentrators.

3.4 Scratch test

The scratch test provides a simple rapid means by which the wear resistance of modified layers can be

Table 1. Plasma nitriding conditions used in specimen

Sample	Nitriding Parameters					
	Temperature (°C)	Pressure (torr)	Time (min)	Ar Flow (sccm)	H_2 Flow (sccm)	N_2 Flow (sccm)
1	440	1.0	180	20	0	50
2	300	4.0	60	20	0	50
3	300	0.60	180	30	20	60
4	300	0.60	180	0	90	30
5	300	0.94	180	0	90	30
6	300	0.90	240	0	90	30
7	350	0.90	240	0	90	30
8	400	0.90	300	0	90	30
9	450	0.90	180	0	90	30

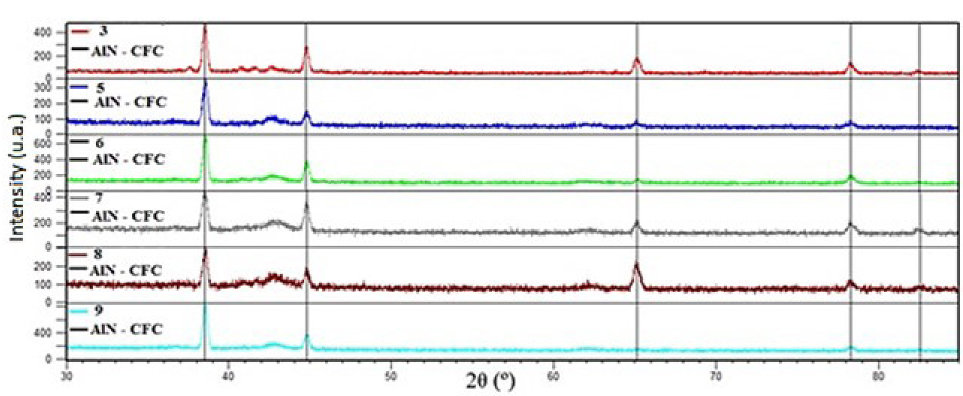

Figure 2. X-ray diffraction (XRD) peaks of the nitriding conditions.

qualitatively assessed. The results obtained for the successfully nitrided samples (conditions 3, 5, 6, 7, 8 and 9) can be seen in Figures 3 and 4, that shows curves of friction force as function of normal force. It was necessary to increase the load applied to 10^5 mN in condition 9 because the nitrided layer was more resistant than the others, so it was necessary to divide the results in two curves.

The mean friction coefficient for the non-nitride sample and for the nitrided samples and the LC, together temperature, pressure and nitriding time of each nitriding conditions are shown in Table 3. The LC (critical load) is the load necessary to remove the nitrided layer and expose the substrate on track [19].

The values μ of the friction coefficient indicate the greater or lesser penetration of the indenter in the material, thus the smaller values μ of coefficient for the nitrided samples suggest better tribological properties in relation to the non-nitrided sample. In nitrided samples the increase in the penetration resistance was observed for longer and heater processes, since there was an increase in the LC, and that is an indication of better tribological properties and wear resistance and this can be related to the higher thickness of nitride layer. Condition 5 presented higher penetration resistance than condition 3, despite had the same temperature and nitriding time, indicating that the presence of argon in condition 3 produces a reducer effect of the resistance. It was observed that conditions 7, 8 and 9 were the most satisfactory, with better resistance, lower values of friction coefficient and higher LC.

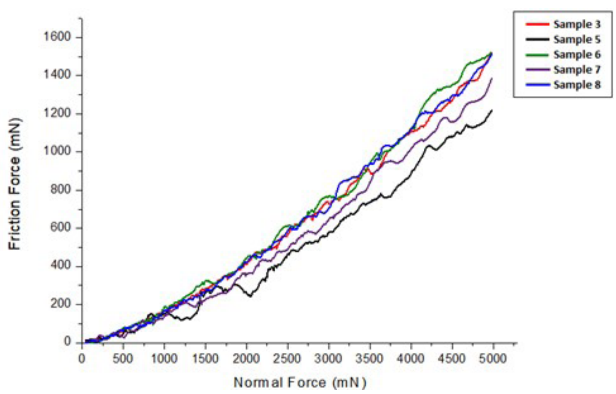

Figure 3. Curves of Friction Force × Normal Force obtained during the scratching test of samples 3, 5, 6, 7 and 8.

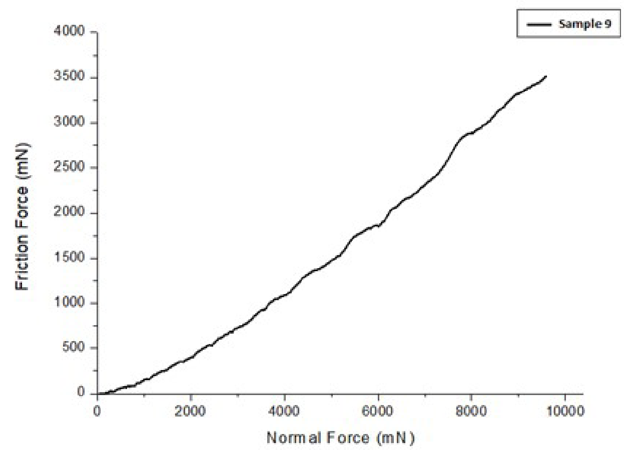

Figure 4. Curves of Friction Force × Normal Force obtained during the scratching test of sample 9.

Table 2. Superficial roughness obtained by mechanical profilometry of nitrided and non-nitrided samples

Sample	Results (μm)			
	1	2	3	Avarage
Non-Nitrided Sample	0.09	0.08	0.07	0.08 ± 0.01
3	0.17	0.57	0.17	0.30 ± 0.23
5	1.03	1.10	1.13	1.08 ± 0.05
6	0.32	0.32	0.34	0.33 ± 0.01
7	2.01	1.91	1.85	1.92 ± 0.08
8	1.72	1.61	1.75	1.69 ± 0.07
9	0.78	1.15	0.72	0.88 ± 0.23

Table 3. Friction coefficients and critical load (LC) obtained in scracthing test associated with nitriding conditions

Scracthing Results			Nitriding Parameters		
Sample	Friction coefficient (μ)	LC (N)	Temperature (°C)	Time (min)	Pressure (Torr)
Substrate	0.49	-	-	-	-
3	0.24	2.55	300	180	0.6
5	0.19	2.90	300	180	0.94
6	0.15	3.00	300	240	0.90
7	0.18	3.80	350	240	0.90
8	0.06	4.00	400	300	0.90
9	0.02	4.00	450	180	0.90

4 Conclusion

The presence of the AlN surface layers, with CFC structure, on the AA7075-T6 substrates by plasma nitriding was verified by XRD. The pressure applied during the nitriding process should not exceed 1 Torr and the increase in power should be slow to avoid the formation of eletric arcs that cause the melt of material. The conditions 3 (0,6 Torr, 300 °C for 180 min), 6 (0,9 Torr, 300 °C for 240 min) and 9 (0,9 Torr, 450 °C for 180 min) were the most satisfactory for obtaining AlN layers with the lowest surface roughness. Scratching test indicates greater surface hardness of the nitride samples compared to the non-nitrided substrate. An increase in the strength of the nitrided samples was observed with increasing time and nitriding temperature, that is an indicative of increase in tribological resistance and wear resistance of the AlN layer. The analysis carried out suggests that among all nitriding conditions the nitriding condition 9 was the most satisfactory, since indicates the presence of a nitride layer with a darker coloration, less roughness, lower coefficient of friction and higher LC.

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